

Article

Washable, Low-Temperature Cured Joints for Textile-Based Electronics

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Abstract: Low-temperature die-attaching pastes for wearable electronics are the key components to realize any type of device where components are additively manufactured by pick and place techniques. In this paper, the authors describe a simple method to realize stretchable, bendable, die-attaching pastes based on silver flakes to directly mount resistors and LEDs onto textiles. This paste can be directly applied onto contact pads placed on textiles by means of screen and stencil printing and post-processed at low temperatures to achieve the desired electrical and mechanical properties below 60 °C without sintering. Low curing temperatures lead to lower power consumption, which makes this paste ecological friendly.

Keywords: conductive adhesive; silver flakes; screen-printing; low temperature; printed electronics



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1. Introduction

Electronics and microelectronics play a huge role in people's lives. Laptops, mobile phones and smart watches accompany us every day. There is a lot of effort from the scientific and industrial side to further make electronics commensurable with new shapes [1,2] and substrates to make it even more functional. One of the main directions of this integration is textile-integrated electronics (e-textiles, wearables) [3]. This type of electronics must maintain the capabilities of classic electronic systems while meeting new, unusual requirements, which include flexibility and extensibility [4–6].

E-textiles are already being tested for use in medicine [7], sports [3] or even for everyday use [8]. One of prospective possibilities to produce textile-integrated electronic devices are methods from printed electronics, in particular ink-jet [9] or screen printing [10] techniques. Using these techniques, it is possible to directly print electronic components such as electrodes [11], sensors [12], electrical interconnects, etc. on fabrics or on polymer-coated fabrics [13]. Additionally, the realization of e-textiles by combining the textile and electronic components with Anisotropic Conductive Film ACF has been reported [14]. Washable screen-printed antennas on textiles have been demonstrated in [15]. Noteworthy is the demonstration of ink-jet-printed graphene–silver composite ink on textiles [16].

Finally, washable graphene-based printed electrodes on textiles for wearable health monitoring devices promise potential applications [17]. As the main issues to overcome, the authors of the above articles mentioned cracking and delamination of the layers. No washable joints have been reported to date.

Albeit a variety of printed stretchable electronic devices have been reported in the literature, there are a variety of issues which still remain unsolved [18–20]. One significant

issue is the challenge to maintain stable electrical performance of the textile integrated device and thereby its components under mechanical deformation. Other important issues are development techniques that are compatible with conventional textile production processes, washing durability and the integration of functional electronic devices into wearable systems [21,22].

In order to shed light into the assembly of electronic components onto textiles, one needs to emphasize following points. First of all, attached systems should not restrict motion; however, the main solution, flexible electronic systems with adequate performance, are still a dream of the future [23]. They also require specialized devices for flip-chip assembly [24] and specially prepared contact fields of the chip itself, containing an additional layer of non-conductive adhesive. Bringing such technology to market will be very expensive, which may not prove feasible for most companies. For now, the focus should be on assembling small systems that increase the performance of e-textile systems without any impact on user comfort in a simple and inexpensive way to implement. However, these systems must meet the basic requirements for all e-textile systems: they must be flexible, stretchable and reliable. Additionally, they should work after cleaning, so they should withstand being put in a washing machine with detergents.

In this work, we developed electrically conductive silver-based pastes for attaching LEDs and resistors to fabrics. We applied stencil printing [25] techniques to print the paste on textiles. The stencil printing technique was selected because it is an easy to adapt, industrially proven, a cheap process and it is compatible with the conventional roll-to-roll [26] processing of textiles.

The authors fabricated low-temperature processible silver-based pastes for die attachment with good electrical stability. Resistors and LEDs were attached onto a 100 × 100 mm cotton jersey textile and tested electrically before and after multiple washing cycles. Furthermore, the authors conducted mechanical stretching tests up to 1000 cycles and bending tests up to 10,000 cycles. The electrical contacts were characterized using X-ray microscopy, Scanning Electron Microscopy (SEM) and Electron X-ray Diffraction Spectroscopy (EDX) techniques and optical microscopy.

The focus was put on fabricating a paste for attaching the resistors and LEDs at lower temperatures, below 60 °C, to avoid the degradation of textile substrates. To our knowledge, no comparable pastes for die attaching have been reported to date.

2. Materials and Methods

2.1. Thermal Transfer Contact Pad Preparations

The silver contact pads were made by transfer screen printing methods. Thermal transfer printing is a type of transfer printing commonly used in the textile industry to apply layers to textile substrates. This indirect printing technique uses plastisol inks and special substrates in the form of foil or transfer papers. It allows the application of a mirror image of a multi-layer pattern previously printed on a temporary substrate [23] onto a textile substrate. Authors in earlier works developed a polymer resin based on thermoplastic polyurethane (TPU) and used it to develop a silver composition with a high mechanical tensile strength [8].

As the functional phase, to develop a high conductive composite, silver flakes AX 20 LC from Amepox Company (Łódź, Poland) with average particle sizes of 2–4 µm were used. A printable composition containing 75 wt.% silver flakes in 15 wt.% solution of TPU in dimethylformamide (DMF) was applied through the 77 T screen. After curing at 130 °C for 15 min, a layer of plastisol glue was applied to the silver layer, and then the contacts were deposited onto a textile substrate using the thermal transfer method. The process was conducted in an air atmosphere.

2.2. Joining Material Application

The pastes designed for joining were applied with screen printing, stencil printing and with a needle dispenser. To join LEDs and resistors, die attach pastes were fabricated as

described as follows: silver flakes of two different types, AA-4077 (PSD90 of 15.6 μm) and AC-6652 (PSD90 of 1.7 μm), were supplied by Metalor Technologies SA (Marin, Switzerland). Fugitive rheology additive and dispersing agent, 3,5-Dimethyl-1-hexin-3-ol, DBE-9 (Dibasic Esters) were purchased from Sigma Aldrich (St. Louis, MI, USA). Polymer slurry Noriphan and solvent-based additive F013 were purchased from Proell GmbH (Weissenburg, Bayern, Germany). The silver flakes were added to the slurry at a concentration of 50% to 80%, where the relative amount of each silver flake type compared to entire amount of silver in the formulation was 75% of AC-4077 and 25% of AA-6652. The ratio of 1:3 was chosen based on our previous work [27] and the study published in [28]. Subsequently, after adding the silver flakes to the Noriphan, the slurry of DBE-9 was added, and in the following step 3,5-Dimethyl-1-hexin-3-ol and subsequently Solvent F013 (Proell GmbH, Weissenburg, Bayern, Germany) were added. The entire formulation was speed mixed at 800 rpm for one minute at room temperature. Note that the paste formulations were labelled VJ 50, VJ 60, VJ 70 and VJ 80 corresponding to the concentration of silver particles in the formulations of 50%, 60%, 70% and 80%, respectively.

VJ pastes' dynamic viscosity was tested using the Brookfield RS CPS+ cone-plate rheometer. The pastes containing 50, 60, 70 and 80% of the functional phase remain within the range of 10–30 Pa·s, implicating that the designed pastes are suitable for screen and stencil printing.

For current measurements, VJ pastes were applied with a needle dispenser onto the contact pads prepared earlier on the cotton substrates. SMD 0805, 0 Ω resistors as well as 0603 LEDs were joined onto the contact pads with previously applied pastes and cured for 30 min at 50 °C. Next, a drop of TPU for encapsulation was applied with a needle dispenser and cured for another 30 min. Schematic is shown in Figure 1.

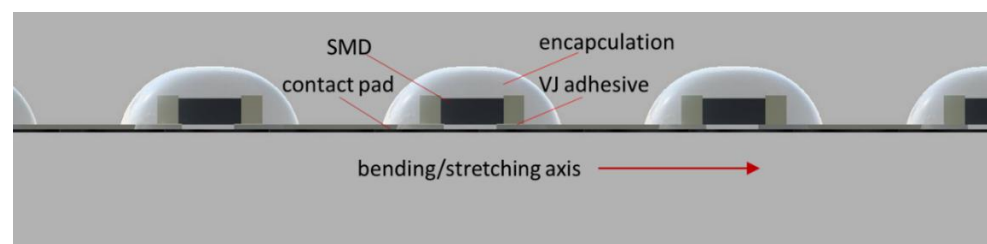


Figure 1. Schematic of diodes/resistors mounted on textile substrate.

2.3. Electrical Measurements

Resistivity was calculated on the basis of measured sheet resistance of layers printed on polycarbonate layers. The final resistivity after 30 min curing at 50 °C for VJ 60 was $1.3 \times 10^{-6} \Omega\text{m}$, $1.0 \times 10^{-6} \Omega\text{m}$ for VJ 70 and $6 \times 10^{-7} \Omega\text{m}$ for VJ 80. What is worth mentioning, after 30 min curing at 90 °C, VJ 70 resistivity drops to $7.2 \times 10^{-7} \Omega\text{m}$. Joint resistivity may differ from layer resistivity. That is why we decided to focus on joint resistance and its change after mechanical tests.

The samples for electrical, bending and stretching measurements were prepared on cotton substrates with contact pads. Chains of five resistors in line were attached to the contact pads. Prior to mechanical measurements, all of the samples were electrically measured using a Keithley 2001 multimeter. Additionally, LED chains were made in the same manner as resistors.

2.4. Mechanical Shear Strength Measurements

Measurements of the shear-strength resistance of joints were made on a two-column Comtech testing machine with a 500 N sensor, where the shear clamp is attached to a strain gauge, and the force at which shearing takes place is recorded on a computer. The accuracy was $\pm 0.1 \text{ N}$.

Substrates and dummy dies were prepared using 1 mm-thick polycarbonate. Substrates were 10 × 10 mm and dummy dies were 2 × 2 mm. Prior to joining, surfaces were

cleaned with acetone, then contact pads were stencil-printed, with layer thicknesses at 40 μm prior to attaching the dummy dies. Curing was conducted with the same parameters as for the cotton samples: 30 min curing at 50 $^{\circ}\text{C}$ for pastes and an additional 30 min of curing at 50 $^{\circ}\text{C}$ for samples with additional TPU encapsulation.

2.5. Stretching and Bending Tests

Stretching and bending tests were conducted with a Comtech testing machine as well. Cotton samples with encapsulated 0 Ω resistors and diodes were used. A total of 1000 stretching cycles of the textile samples were carried out along the mounted elements with a speed of 30 cycles/min and a final elongation of 110%. Electrical measurement of the resistor chains was conducted after 100, 300, 500 and 1000 cycles. Diodes were tested both one by one and all at the same time.

To test the bending reliability, 10,000 cycles were conducted, with the radius changing from ∞ to 5 mm and a speed of 60 cycles/minute. Electrical measurements were conducted after 1000, 3000, 5000 and 10,000 cycles.

2.6. Washing Machine Tests

To perform the washing machine test, 8 samples fixed onto double-layered plain woven cotton swatches (200 \times 200 mm) were prepared. Each sample contained five 0603 SMD diodes mounted with VJ 60 and five diodes mounted with VJ 70, all of them encapsulated. Four such samples were put into a protective washing bag, typical for washing delicate laundry items. The other four samples were put directly in the machine.

The washing machine used for the tests was an Electrolux WH645, an ISO 6330-compliant machine (type A in the standard). The washing program was compiled using Electrolux Laundry Program Manager LPM 6. It is modeled after a household washing program for silk/delicates. The structure of the program is:

- main washing phase—25 min, 40% on-time (percentage of time when the drum is turning), water volume: 12 L,
- intermediate spinning (2 min, 500 rpm),
- 2 \times rinsing (cold water, 40% on-time, 12 L),
- final spinning (5 min, 800 rpm).

The washing temperature during the main washing phase was 30 $^{\circ}\text{C}$, with a total program duration of around 40 min (+/−2 min). Samples were air dried overnight. The detergent used for the tests was Frosch liquid wool detergent (40 mL). For a total load of 2 kg, polyester-based load items were added to the samples, in accordance with ISO 6330.

3. Results

Figure 2a depicts X-ray microscope images of joined resistor elements on textile. Furthermore, an SEM image of the cross-section of a resistor element joined with a VJ 60 paste (Figure 2b) with the corresponding EDS material composition map (Figure 2c) is presented.

The electrical resistance after mechanical stretching and bending measurements is shown in Figure 3a,b. Resistor chain resistance after mounting was the lowest for VJ 60, containing 60% of silver particles and significantly higher for VJ 70 and VJ 50 containing 70% and 50% of silver particles. In the case of stretching (Figure 3a), the largest increase in resistance after 1000 cycles was measured for VJ 70 with 26%, from 3.11 Ω to 3.92 Ω . For VJ 60, the resistance changed from 0.95 Ω up to 1.01 Ω , which is an increase of about 6%.

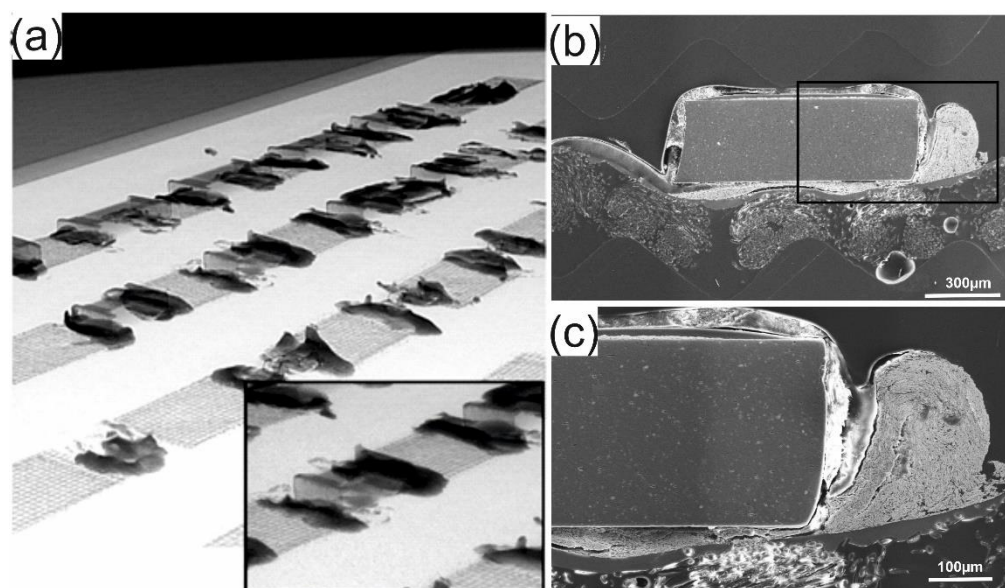


Figure 2. X-ray micrographs of resistors picked and placed on textile (a), SEM-micrographs of the cross-section of a resistor on textile (b). Zoom-in of the black framed region in (b) of the contact area resistor textile (c).

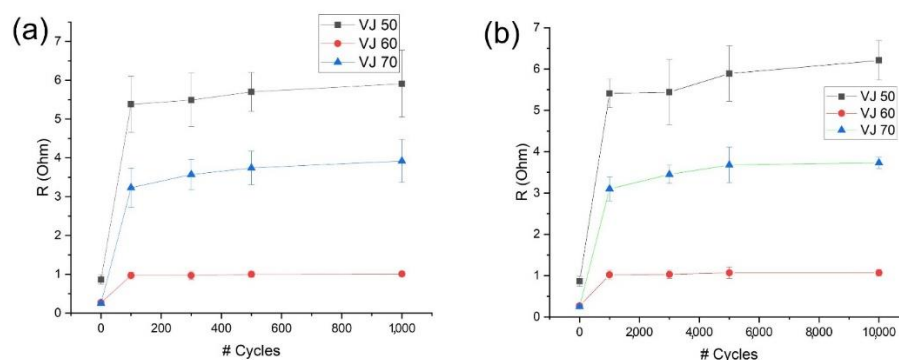


Figure 3. Dependence of the electrical resistance of resistors chain on (a) number of stretching and (b) bending cycles.

For the bending tests, the biggest change was an increase of 20% for both VJ 50 and VJ 70 after 10,000 cycles. For the best conducting VJ 60, the resistance rose by 14%. It is worth mentioning that none of tested samples stopped working during the cycle testing. Additionally, samples with attached LEDs were tested and none of them stopped working.

When looking at the shear strength of joints prepared with the VJ pastes, the smaller the silver content of the paste, the higher the shear strength, as shown in Figure 4. The highest shear strength without encapsulation, 3.7 MPa, was observed for VJ 60, while VJ 50 had 3.4 MPa. Joints created with pastes containing more silver particles had significantly lower mechanical parameters. For comparison, a commercial conductive epoxy adhesive, labeled as CH (Figure 4), was used, with joints cured at the recommended 65 °C for 20 min. According to the data sheet, the shear strength should be 6.5 MPa, while the measurements were allowed to achieve 4.1 MPa, 10% higher than VJ 60. It should be noted that the commercial conductive epoxy's resistivity shown in the data sheet is an order of magnitude worse compared to the VJ pastes created by the authors.

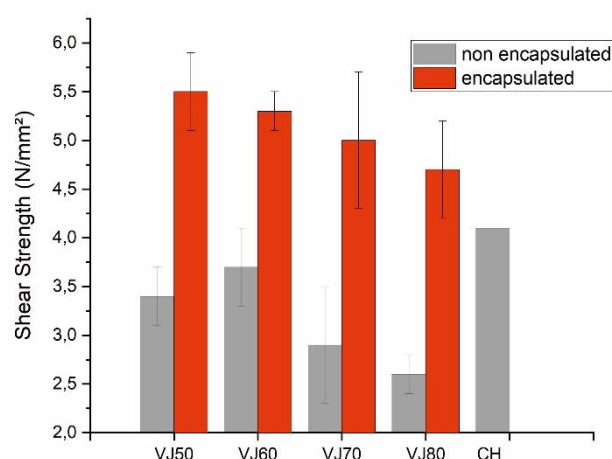


Figure 4. Shear strength of raw and encapsulated joints.

To improve the shear strength resistance, a drop of TPU was added as encapsulation (as shown in Figure 1). The addition of TPU increased the shear strength resistance of the joints by about 2 MPa, as shown in Figure 4. This method resulted in an increase in the joints' shear resistance for VJ 50, 60 and 70 rose to above 5 MPa, achieving the highest value of 5.5 MPa for the joints made with VJ 50.

The role of TPU, apart from improving shear strength resistance, was to protect mounted SMDs during washing in a washing machine. Up to 10 washing cycles were conducted for VJ 60 and VJ 70; results are shown in Figure 5. Conductivity was checked after the 1st, 3rd, 6th and 10th washing cycles. The first washing cycle did not impact the samples at all. After the third cycle, the first LEDs—mounted with VJ 70 and washed without the protective bag—fell off. Two LEDs, mounted with VJ 60 and VJ 70, both washed without the protective bag, stopped working, which means the connection between the chip and the pad broke. After six cycles, another LED detached. At this point, the first of the SMDs washed in the protective bag stopped working as well—but none of them fell off.

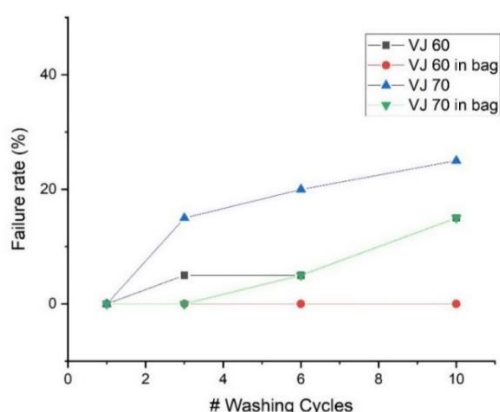


Figure 5. Life of diodes versus number of washing cycles (two types of washing, two pastes).

After 10 cycles, the highest failure rate (exceeding 20%) was observed for VJ 70 without the protective bag. For the VJ 70 samples in the protective bag and the VJ 60 samples without the protective bag, the failure rate was similar at about 15%. All of the diodes mounted with VJ 70 and washed in the protective bag were still attached. All of the diodes mounted with VJ 60 and washed in the protective bag worked properly, achieving a failure rate of 0%.

SEM pictures of cross-sections from the textile samples were made to determine the state of the joints after 10 washing cycles. Figure 6a,b show properly conducting VJ 60 joints in a side view, after 10 cycles of washing tests in the protective bag. The joints show

no delamination and no cracks. For comparison, in Figure 6c,d, non-conducting VJ 70 joints after 10 test cycles in the washing bag are shown. The conductivity was lost due to the delamination shown in the picture, without detachment of the LEDs from the substrate. The delamination (shown in a red circle) shows that the connection between the chip and the contact pad is broken after the washing cycles.

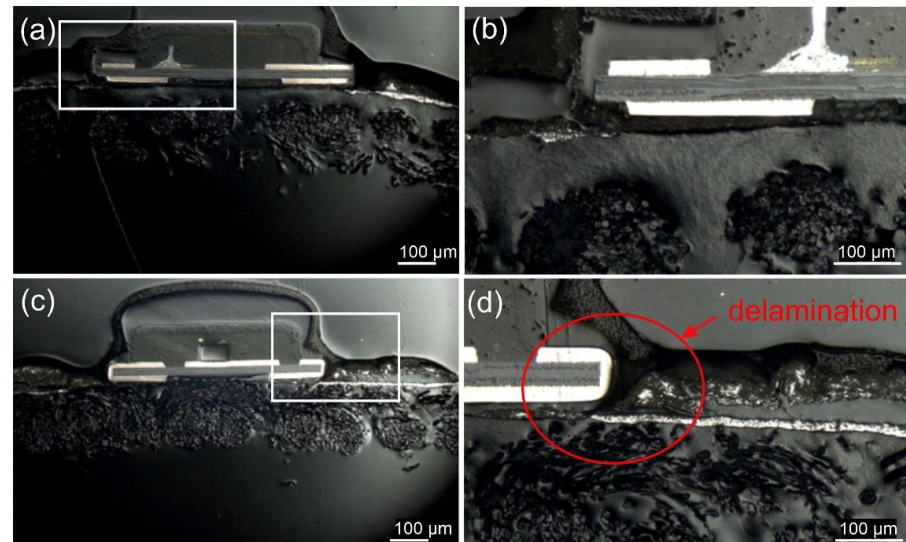


Figure 6. Cross-section SEM image of a properly conducting (a,b) and delaminated joint (c,d) after 10 washing machine cycles.

Figure 7 depicts optical images of the cotton textile with die-attached LEDs and resistors to illustrate the effect of mechanical bending and creasing of the textile. In Figure 7a, working LEDs upon applying voltage are shown. Furthermore, the LEDs remain lit up upon bending of the textile Figure 7b. After creasing the textile, see Figure 7d (inset), the LEDs maintain their function (Figure 7e).

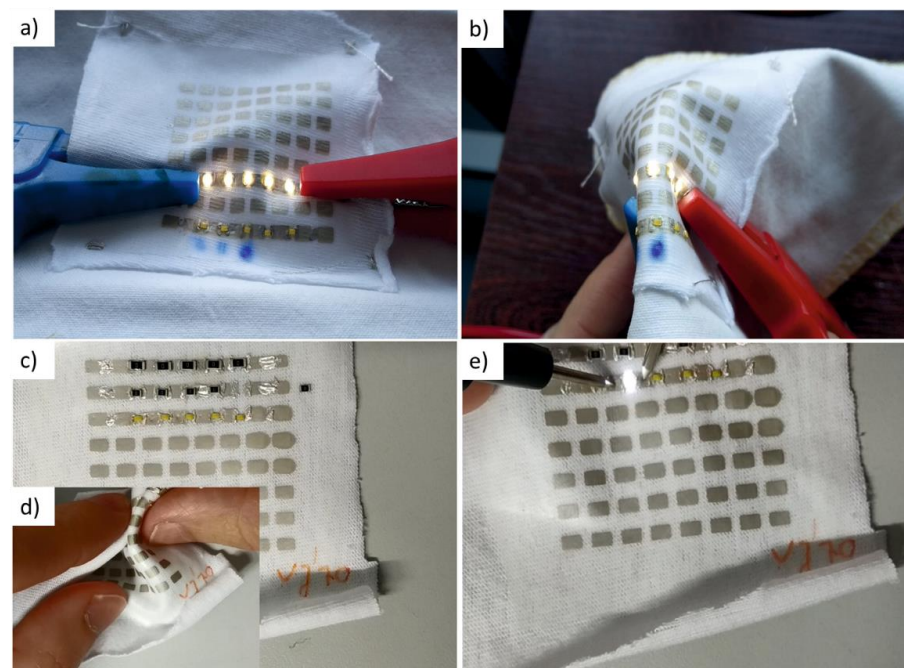


Figure 7. Optical images of textile with die-attached LEDs and resistors before bending (a), during bending (b), before crease (c), during crease (d) and after crease relief (e).

4. Discussion

The first aspect that draws attention is the much lower resistance of the assembled systems with the use of VJ 60 paste, containing 60% of the silver flakes, compared to that of the systems with joints containing 70% of the silver flakes, which is surprising. In the case of a paste containing 50% silver, everything follows the theory of percolation. This shows that with an increase in the content of conductive particles, the number of connections between particles increases, which translates to an increase in the conductivity of the layer. Thus, the highest resistance was obtained for the paste with the lowest content of conductive particles. The lower resistance of connections made with VJ 60 may be explained by the use of two sizes of silver flakes in the preparation of pastes. A total of 75% of the entire conductive material consists of larger flakes with a diameter exceeding 10 μm . Considerable densification of such material, in combination with covering the printed joining layer with the installed chip, may cause less evaporation of solvents during curing. Moreover, the larger amount of residual solvents within the printed joining layer could result in increased swelling of the binder polymer, which in turn would also decrease the percolation between conducting silver flakes. The aforementioned effects could both result in higher resistance of joints made with VJ 70 paste.

Measurements taken when stretching the samples up to 110% of the initial length showed that regardless of the VJ paste variant used, we obtain an increase in system resistance not exceeding 30% of the initial value, as shown in Figure 3. The results obtained with VJ 60 containing 60% silver are of particular interest. In this case, the low increase in resistance is probably the result of the appropriate ratio of the polymer to the particles of the conductive material. The right amount of polymer allows the flakes to be better held together while maintaining the flexibility of the joint. In the case of a combination containing 70% silver, micro-cracks or delamination of the layer occur more easily due to the smaller amount of polymer.

Bending resistance measurements show the change in the resistance of the system not exceeding 20% (Figure 3), and again the smallest change was noted for the VJ 60 paste. This can likewise be justified by the appropriate ratio of polymer to conductive material. The highest stresses during bending, occurring on the edges of the chip, did not exceed the stress resistance of the joint material. Undoubtedly, the TPU encapsulation strengthening the joints helped us to obtain a small change in resistance in all samples, as shown in Figure 4.

Washing tests have proven that under certain conditions, it is possible to wash the systems mounted with the use of the developed pastes multiple times. The protective bag had a significant impact on the number of damaged joints. In the case of the VJ 70 paste, the first non-working LEDs appeared after three cycles, while none of them fell off even after 10 cycles. This means that apart from the notorious bending and creasing (similar to creasing shown in Figure 7) during washing, the mechanical impacts of the diodes against the washing machine drum or their random contact with other washed materials have a large impact on the failure rate. On the other hand, the creasing and bending itself has an impact on the assembled systems anyway, as evidenced by the much better survivability (zero failure rate) of the systems prepared with the use of VJ 60 paste, characterized by better mechanical resistance.

5. Conclusions

In conclusion, we formulated an easy-to-apply joining technology based on designed silver-based conductive pastes. Their electrical properties, accompanied with the adhesion strength are comparable with the top adhesives used on the market. The pastes can be cured at a very low temperature, which is a novelty to date. The technology may be used for joining chips onto textile substrates at temperatures as low as 50 $^{\circ}\text{C}$. In such temperatures, the authors were unable to find materials with such low resistivity. The joints may be stretched, bent and even creased with very low impact on the conductivity of the system.

That implies that the textiles with mounted chips may be washed in the washing machine in a protective bag without any failure.

As a washable technology, the designed material is suitable for joining in complete textile systems for everyday use. With the use of common dispensing, stencil- or screen-printing methods, it is easy to implement, with relatively small financial outlay.

Author Contributions: J.S. and V.S. performed most experiments, electrical and mechanical measurements, final data analysis and wrote the paper. D.J. prepared the substrates and contact pads. S.R. performed the washing tests. L.W. prepared samples for SEM and SEM photographs. C.K. and M.v.K. helped designing the research. M.S.-R. and M.J. put forward some amendments. All authors have read and agreed to the published version of the manuscript.

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